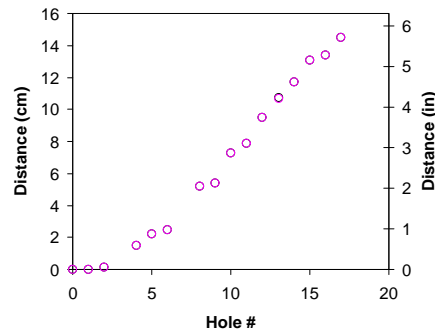
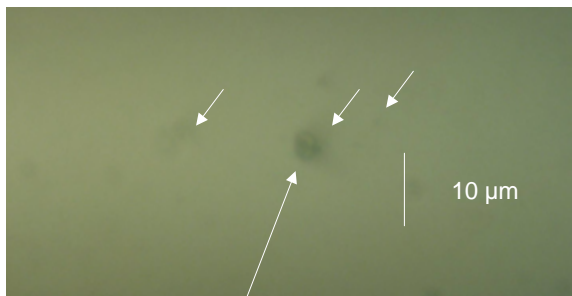


The arrangement of the bubbles and depressions were found to be in a fairly straight line and parallel with the longitudinal axis of the fiber. Figure 14 is a plot of the distribution of the features along the length of one of the “588” samples.



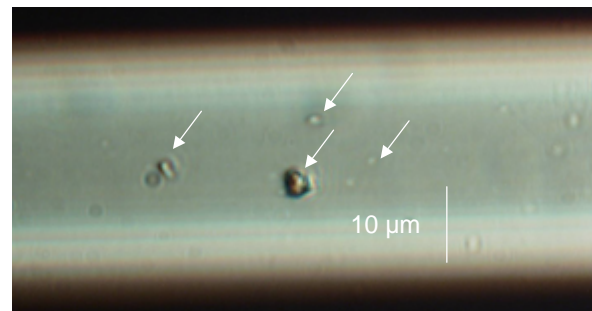
**Figure 14** Distribution of Features Along a 6” Sample of CD0588XD Fiber

Additional work with these samples was done to catalogue their relationship to breaches in the carbon coating below them. Not only were breaches found below some of the bubbles, but others were found in the vicinity of the bubbles but with no bubble on top of them as seen in Figure 15a and b.



*Courtesy of The Aerospace Corporation*

*a. Features Below Polyimide Bubble,  
Reflection Mode 100x, 0.3 μm Resolution*

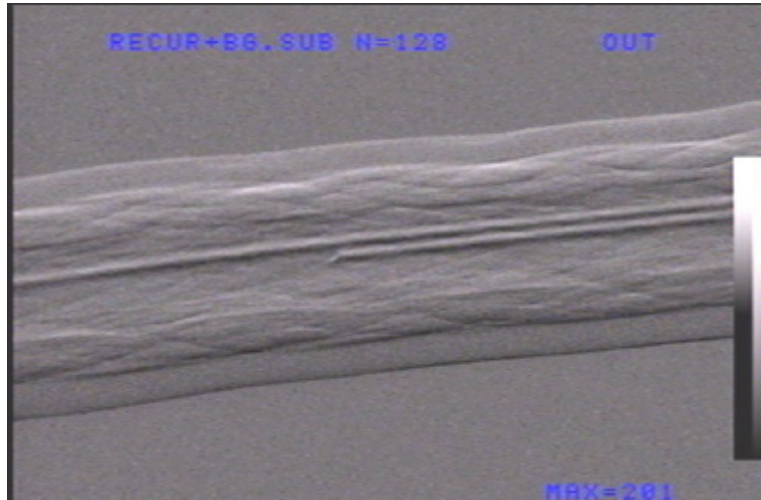


*Courtesy of The Aerospace Corporation*

*b. Polyimide Etched off with Sulfuric  
Acid, Multiple Bright Spots at Carbon  
Coating Surface, Transmission Mode*

**Figure 15** Carbon Defects Seen Below Polyimide on Sample of CD0588XD Fiber

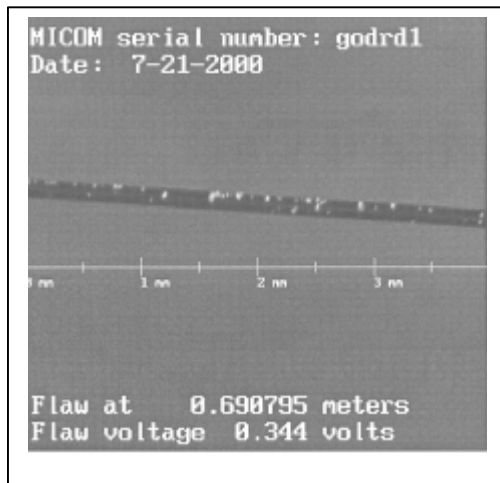
X-ray was used to see if defects on the surface of the coated fiber could be detected. Figure 16 shows that X-ray can be used to look at the fiber without removing the cable elements which is useful for inspecting for fiber which may be too long for the jacketing (in cases where the jacket and/or buffer has shrunk).



*Courtesy of The Boeing Corporation*

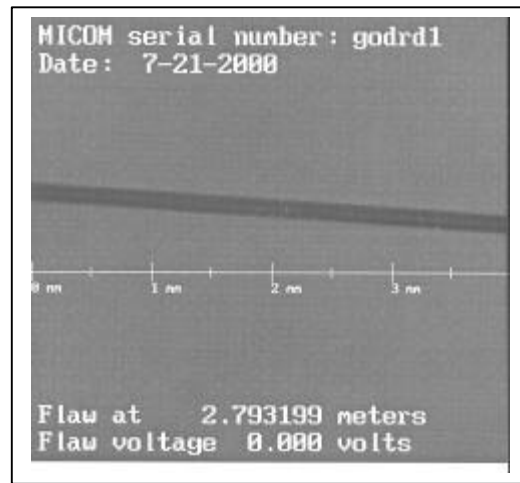
**Figure 16** E. Banks' Sample With a Broken Fiber Whose Ends Overlap

A fiber inspection system, built by the US Army Aviation & Missile Command for their own internal use, was used to try to inspect for 10  $\mu\text{m}$  sized features in the polyimide surface that might indicate the presence of the bubble feature that has been consistently associated with "rocket engine" defects and low strength breaks. The system uses an infrared detector and looks for signal scatter off of the surface at feature locations. The feature is described with respect to the voltage of the returned scattered signal. When a feature is found that corresponds to a minimum signal voltage, the system takes a picture of the corresponding location. The inspection system is very sensitive to any surface feature, especially contamination such as lint and dust (Figure 17). A lint free dust capture stage was arranged around the fiber and the inspection was continued. Figure 18 shows the image of a debris-free fiber with polyimide surface features.



*Courtesy of US Army Aviation & Missile Command*

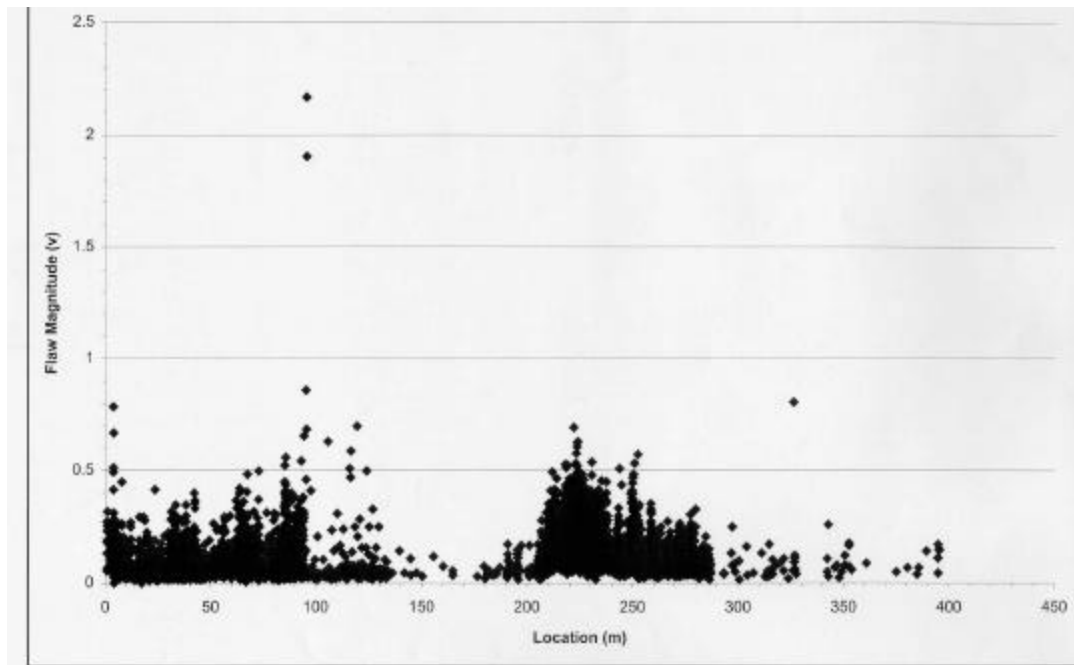
**Figure 17** Debris-Laden Fiber



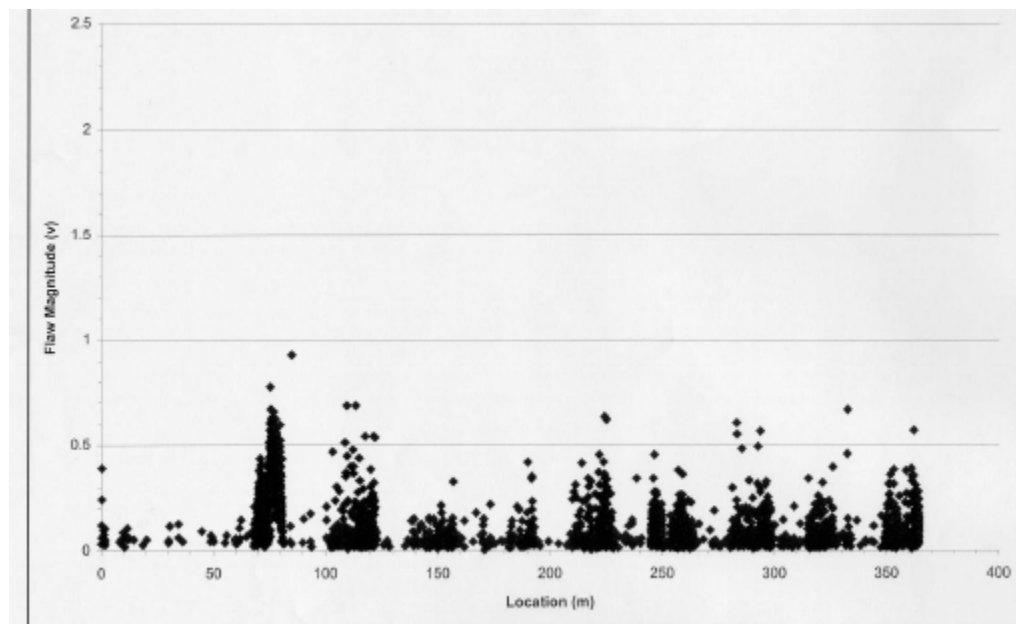
*Courtesy of US Army Aviation & Missile Command*

**Figure 18** Cleaned Fiber

To see if, using a more typical inspections speed, 0.5 m/sec, the machine could provide useful statistics which would differentiate the two reels. Figure 19 and Figure 20 show the results of running approximately 400 m through the machine, looking for a 0.1V feature size or greater. Additional work is required to understand the usefulness of a system such as this to identify defects in the polyimide coating.



**Figure 19** Feature Statistics for CD0588XD



**Figure 20** Feature Statistic for CD0384XC

## 6.0 MATERIAL BEHAVIORS AND INTERACTIONS

The interactions between the materials used for the fiber, its coating, and the cabling components are believed to be at the root of the “rocket engine” defects. These materials were examined, as were their interactions in the environments created by the manufacturing processes. Artifacts found during the DPA’s led the investigation team to hypothesize about various material reactions including: decomposition of the FEP buffer, discontinuities in the carbon/polyimide fiber coating leaving the glass exposed to etchants and moisture, the electrical properties of the carbon/polyimide coating and the thermal stability of the polyimide layer.

### 6.1 FEP Decomposition

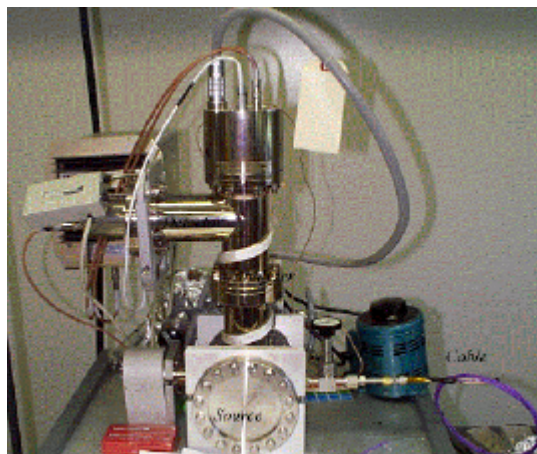
FEP is a copolymer of tetrafluoroethylene (TFE) using hexafluoropropylene (HFP). It is sometimes generally described as a Teflon® however its properties and processing requirements can be very different than those of the homopolymer PTFE. FEP is applied in optical cable manufacture using a melt extrusion process. The maximum service temperature for FEP is 206°C (400°F) and the maximum processing temperature is 400°C (746°F). The temperatures used in the BICC General process extend close to the maximum processing temperature.

Decomposition of FEP depends upon the composition and temperature of the ambient environment. In a moist or dry environment containing oxygen, FEP decomposes at a rate of 0.05% and lower, by weight, below 325°C (612°F) and at a rate of 2.5% and higher, by weight per hour at 400°C (746°F) [ref 9,10, 11]. In a moist or dry nitrogen environment the decomposition rate at 400°C (746°F) drops to 0.40%/hour. When the atmosphere contains oxygen, the decomposition product is mainly COF<sub>2</sub>. In a dry, nitrogen atmosphere COF<sub>2</sub> is not created.

Though the COF<sub>2</sub> was the majority decomposition component measured by Baker and Kasprzak, they noted that it was difficult to capture all of it in the measurement because it so rapidly reacts with air, water and the quartz chamber the FEP sample was heated in. Baker and Kasprzak remind us that at the temperature and humidity (50% RH) of their experiments COF<sub>2</sub> “hydrolyzes considerably according to:”



Testing was performed by NASA GSFC to confirm the evolution of reactive, fluorine containing gases, such as HF or COF<sub>2</sub> from the NFOC-2FFF-1GRP-1 cable due to mechanical agitation and exposure to an electric field. The set-up is shown in Figure 21. The NFOC-2FFF-1GRP-1 cable and OC1260 cable were tested. The OC1260 cable uses the same cable materials as NFOC-2FFF-1GRP-1 except that its optical fiber does not have the carbon layer under the polyimide coating. In both cases, the length of cable was hooked up to a mass spectrometer. After pumping out all the gasses for between 16 and 72 hours, the cable was stressed in the two different ways, mechanically twisting and exposure to an electric field.



*Courtesy of NASA GSFC*

**Figure 21** Set-up for Mass Spectrometer Measurements of Fluorine Species Released From Fiber Optic Cable

Mechanically twisting the NFOC-2FFF-1GRP-1 cable resulted in the release of fluorinated species that persisted for about five minutes. Mechanically twisting the OC1260 released three orders of magnitude less Fluorine. The spark from a Tesla coil was run over a length of the cable to induce charge breakdown by the cable components and the polyimide coating ending at the grounded carbon coating in the NFOC-2FFF-1GRP-1 cable. This condition also resulted in a release of fluorinated materials, in larger quantities, lasting again about five minutes. In the case of minimal air leakage into the cable, a longer distance between the spark and the mass spectrometer corresponded to a lower mass flux rate from the cable and a longer mass flow. After 48 hours, a significant fraction of the gasses entering the mass spectrometer was water.

## 6.2 Polyimide Related Issues

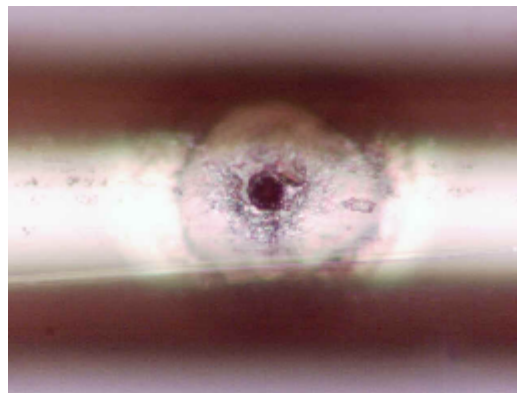
The polyimide coating is intended to protect the fiber from mechanical insults. Its operating temperature range allows it to perform this function from  $-269^{\circ}\text{C}$  ( $-452^{\circ}\text{F}$ ) to  $400^{\circ}\text{C}$  ( $752^{\circ}\text{F}$ ). This range makes it suitable for use by ISS external vehicular applications where coating materials normally used for commercial applications would fail. The polyimide coating also plays a role with respect to damage that may occur to the carbon layer and the glass when the fiber is exposed to a static electric field and how HF generation may be occurring. DPA's of the "rocket engine" defects showed that the polyimide layer at these defect sites often contains a void (Figure 7). It is not known how these voids occur, though they are consistently associated with the rocket engine defects, and the low strength defects. They have been found in coated fiber obtained directly from Lucent-SFT indicating that they are created prior to shipment and processing by SEA or BICC. Their root cause and their role in the reduction of fiber strength following the many processes used at BICC is not yet understood.

The polyimide is applied to the fiber through a polymerization process, not a melt process. DuPont's recommendations for fully polymerizing polyimide could not be located. We were however, able to find the glass transition temperature ( $T_g = 325^{\circ}\text{C}$ ), the dielectric withstanding voltage ( $\text{DWV} = 4 \text{ kV/mil}$ ) and the modulus of the material used on this fiber ( $245 \text{ kg/mm}^2$ ) [ref 12]. Polyimide should not melt when its glass transition temperature is exceeded. Lucent-SFT does not exceed the  $T_g$  when it processes the polyimide coating. Though Lucent-SFT utilizes cure temperatures that are recommended by DuPont to drive off the solvents, the rapid travel speed through the cure ovens may not allow complete solvent outgassing.

Polyimide's hygroscopic nature provides a sink for ambient  $\text{H}_2\text{O}$ . This  $\text{H}_2\text{O}$  can, in the presence of  $\text{COF}_2$  at room temperature and above, result in the creation of HF during manufacture of this cable.  $\text{COF}_2$  and HF can diffuse into polyimide. Polyimide will act as a sink for HF as it does for  $\text{H}_2\text{O}$ . Diffusion of HF into and within the polyimide will continue until equilibrium is reached. HF will diffusion into some species of carbon and not into others. If the amorphous carbon layer directly under the polyimide has a species, such as carbon black, which absorbs HF, then the carbon black will be part of the equilibrium dynamics. Some species of carbon do not absorb or react with HF and so will not be part of the HF sink. Bare  $\text{SiO}_2$  or  $\text{GeO}_2$  react with HF and so, are also part of the HF sink in the polyimide/carbon coated glass system. The HF will diffuse into the polyimide as long as there is more HF outside of its outer diameter than there is inside of its volume, plus the volume of the absorbing carbon species plus the amount not yet consumed at the bare glass interface.

Polymers used in electronic packaging have been tested to identify the risks associated with trapped water when the part is suddenly exposed to high temperatures, such as happens during soldering [ref 13,14]. Plastic encapsulated microcircuits have been found to fail due to delaminations at the plastic-to-metal lead frame interface when the trapped water expanded. These type of failures, known as popcorning, were found to occur at material interfaces rather than within the homogeneous material, even if voids were present. Better adhesion of the materials by the use of a primer or by roughing the surfaces is reported to be possible solutions for reducing water collection at these interfaces. Popcorning as a root cause has not been investigated as of the date of this report.

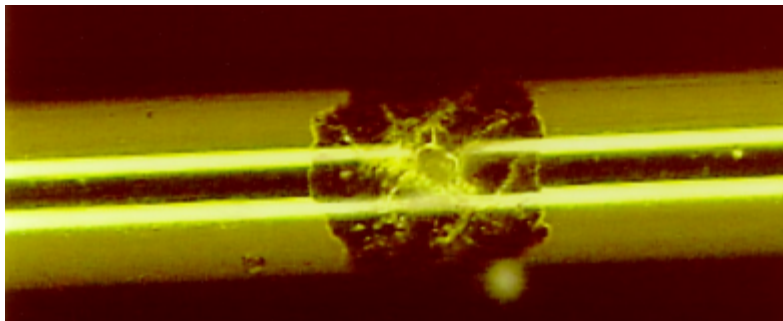
The final thickness of the polyimide on the BF04515 is  $15\text{ }\mu\text{m}$  (0.60 mils). The cure schedule used may make its mechanical and electrical characteristics differ from DuPont's published values [ref-12]. The polyimide coating on a sample of BF04515 fiber, which had never been cabled, was tested for dielectric strength. Several lots were not tested so we do not know how the value may vary on a lot basis. A value of 6.5 kV/mil in 50% humidity [ref-15] was measured (See Section 6.5 below for the test set-up and more discussion about ESD testing). DuPont's published value for fully cured polyimide is 4.0 kV/mil at 50% RH. Burn marks were found on the sample tested (Figure 22).



*Courtesy of The Aerospace Corporation*

**Figure 22** Burn Mark From DWV Testing of "Rocket Engine Defect"- Free Fiber

Another test was done on fiber that was removed from NFOC-2FFF-1GRP-1 cable that was known to be defective (several “glows” found along its length, at least one of which was DPA’d and found to be a “rocket engine”). A voltage of 1.5 kV was enough to break through the polyimide between two electrode locations and sustain a current down the carbon coating between them. A burn-mark was created when the voltage was raised to 2.5 kV (Figure 23).



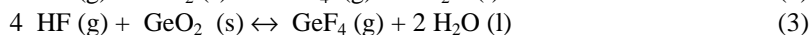
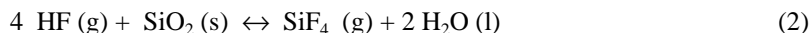
*Courtesy of NASA GSFC*

**Figure 23** Burn Mark From DWV Testing of Fiber with “Rocket Engine Defects”

### 6.3 HF Etching

HF is known as a “weak” acid. It is not significantly ionized in water. As such, it is not reactive with polyimide or most other organic materials. However, due to the polarity and hydrogen bonding characteristics of HF, it has a tendency to absorb readily into and coordinate with the polyimide. Analogous to polyimide’s tendency to retain water, HF is also retained by the polyimide. The polyimide acts as a sink for the HF. When HF becomes part of an etching reaction, more HF is delivered from the surrounding polyimide to the etching site in order to maintain chemical equilibrium.

Hydrofluoric acid is a well-known etchant of glass. The reactivity and reaction product of hydrofluoric acid with glass was first described in a scientific journal in 1771 by Scheele. The formation of germanium tetrafluoride was documented by C. Winkler in 1886. The formulas describing the reaction in the Ge doped fiber are:



The oxides are solid and ordered in the original fiber structure. Mechanistically, the reaction of hydrofluoric acid with a silica surface (or in similar manner with a germania surface) first requires the insertion of water into a silicon oxygen bond. This insertion hydrates the silica, providing a readily exchangeable group to react with the hydrofluoric acid. This step of the reaction is controlled by the activities of the hydronium ion and the silica surface. As the silica is hydrated, the hydroxyl groups present on the silica are replaced with fluoride groups in an extremely exothermic reaction. The rate of the reaction is dependent upon the equilibration of a wide variety of species both as the products and the reactants. Due to the complexity of the reaction, there have been few or no definitive studies of the reaction of HF with siliceous materials in spite of the great interest and long time elapsed since the discovery of the reaction.

Silicon and Germanium fluorides are gases and take up almost 840 times more volume than the oxides, possibly causing a pressure “eruption” of gases out of the orifice. The gas phase nature of the reaction products provides for rapid mass transport of the products from the reaction sight. This minimizes the build up of reaction products that could result in slowing of the reaction. The gas phase reactants will also react in the reverse reaction at a significantly higher rate due to the faster mass transfer rate. Additionally, the